App. F. IX-4

1926-27.

File No. 92-26

Previous References (Genl)

Serial Nos. 1-5

Later References

1. W 38-38 (1-3)

Reserve

(44)

General and Revenue Secretariat.

Serial Nos. Genl

Part B for Sept 27

SUBJECT

Proposal to manufacture chromate in iron works

Ecl. 92-21 (1)

NOTES OF DISCUSSION WITH MR. E.E. DUTT (6-1?

Present.

The Second Member of Council.

Mr. E.E. Dutt,

Mr. B. Jayaram, Director of Geology,

Mr. P. Sampath Iyengar, Professor of Geology

Mr. A.M. Sen,

Mr. C. Ranganatha Rao Sahib, Secretary to Government, Development Departments.

4 & 80
GEN. & REV. SECRETARIAT,
7 JAN. 1927
DEVELOPMENT BRANCH.

---

(1) The Second Member of Council stated that he was pleased to introduce Mr. E. E. Dutt, an Industrial Metallurgist of Europe, to the officers of the Geological Department. Mr. Dutt was in touch with the up-to-date industrialists of Europe and America and would be able to give the Department information on the market and the industrial possibilities of ores and other minerals hitherto considered as not very remunerative, and perhaps also would be able to answer questions by the departmental officers in regard to markets for the products available in the State.

* * * * *

2) (8) Manufacture of cyanide chromates.

Mr. Dutt explained that he had obtained a patent from a French firm for the manufacture of chromates and cyanide by means of a process similar to the Thermit process in which all the heat required was evolved in the course of the chemical re-action. He had submitted a proposition to the Mysore Government and stated that, if it was approved, he was in hopes of being able to manufacture cyanide in the State in quantities sufficient to supply the Kolar Gold Fields.

Extract

WD 01229—GPB—37,500—1-9-26
App. F. III—2]

General and Revenue Secretariat | Revl File No. ~~25~~ 92 of 1926.

Serial No.

Extract of members remarks.

3) We may now take action on information which has just been given.

(Sd) W. W. H.

Secy's note.

4) Please take action on the Peng and on the note of discussion as directed by the member.

(Sd) C. R. S.

Subd

5) Separate action re 'manufacture of cyanide chromates' is being taken in the Land Works Section.

20/1/27. 6) This may be linked to that file and resubd.

WD 11/1

9.6. Section

7) The file refd. to (P.W. 26-26) is linked. ~~Orders are reqd. in the linked file above~~ The linked file was ordered to be Subd. after issuing the letter to the P.W. Secy re Source for meeting the expenditure in accdt. of Mr. Dutt's engagement.

17.1.27 8) Subd for perusal with the linked file.

Sec'y (D) WD 17/1

18/1 9) What is the probable amount involved Perhaps it may be allotted to the sum of Rs 60,000 sanctioned for mining arsenic ore as the Expt is intimately connected with the utilisation of Chromium 19.1.27

WD 19/1

NOTE—Contn. Sheet

WD 01229—GPB—37,500—1-9-26
App. F. III—2]

General and Revenue Secretariat

Genl File No. 92 of 1926.

Serial No.

Refee: — Paras 9 of Note.

10) The ~~estimate~~ probable amount will be as follows: —

1) Fee to be paid to Mr. Dutt for 2 weeks @ Rs. 50/- per day — Rs. 700/-

2) Travelling expenses to & from Bombay to Bhadravati & back — about 300/-

1000/-

11) In case there is any possibility of the continuance of the period of the amount may be more.

12) May the refee: called for from Secy. Board of Management, Mysore Iron Works be awaited. for orders.

26-26

13) Mr Dutt's experiments at Bhadravati are likely to give reliable data: the further report from the Secy I. Works may perhaps be awaited.

Secy D)

25/1

14) 21/1/27 informing Govt ~~Board~~ in Dutt has no working knowledge of this investigation

15) Maybe calculation Engrs

16) Please see marginal note

Engrs 26.1.27

26/1

17) Draft put up.

18) 27/1/27 Appd. Issue

28/1

19) To, Secy. to Management, Ir. Works.

No. 6243 Dy. 2/2/27

Calls for a report of the results of experiments made by Mr. Dutt at Bhadravati

NOTE—Contn. Sheet

WD 01229—GPB—37,500—1-9-26
App. F. III—2]

General and Revenue Secretariat

Geol File No. 92 of 1926

Serial No. 3

From the Secy. Board of Management Sivaganga Works.

No. B. 327/27 & 12.3.27.

Forwds. copy of Ref. Dutta's report on the experiments carried out by him at Bhadravati and copy of a note by Dr. S. S. Dyer, Chief Chemist who continued the experiments after Ref. Dutta left Bhadravati (19.3) in forwarding copies of the report &c. the Secy. Bd. of Management, Sivaganga Works, states that the results of the experiments made by Dr. S. S. Dyer are very disappointing & further opinion has been called for from Ref. E. E. Dutta.

DEWAN'S OFFICE. Received on 16. MAR. 1927 Ref No. 6660

Secy's note. Subd. to 3rd member (20) 21.3.27 14/3

OFFICE OF THE SECOND MEMBER OF COUNCIL. 15 MAR. 1927 No. 1044

20. 15/3/27. Subd for perusal 3rd M. 15/3

W. Secy - Please submit to Dewan also.

22) The report may be sent to the Dy Geology for perusal as

Dewan)

22) 16.3.27

16/3

16/3

16/3

NOTE—Contn. Sheet

6748 5868 5

WD 01229—GPB—37,500—1-9-26
App. F. III—2]

General and Revenue Secretariat | Ecl File No. 92 of 1926

Serial No.

W/ Draft put up.
20/3-27 [signature] 25/3 Issue WP 23/3

4. To,
Branch Entry
No 8730 of 26/3/27
Forwarding reports recd. above for personal return.

From A

From — do — No. D. Dis 12/26-27 of 12/4/27
Returns the records sent with U.O. (4).
26) Secy: Distt report + Copy of a note by Secy. S.S. Depr sent as to the Dist of Ecl. as per orders of the 3rd member (para 22 of Rules) have been returned
27) may the receipt of further information called for from Secy. Distt by the Secy. Bd. of Management, Imam Works. regd. in para 2 of this letter of 12/3/27 be awaited for orders

20/4/27 [signature] Sec'y. D) WP 26/4

The Sec'y Works may be reminded about this by the end of this month.
Yes
9/5 WP 8/5/27

WP 8/5-27

GWB file 28/8 mark

GEN. & REV. SECRETARIAT
9 MAY 1927
DEVELOPMENT BRANCH.

WP 9/5

Reminder issued.
No. D. 2419 of 5-5-27

NOTE—Contn. Sheet

1982 6

WD 01228—GPB—37,500—1-9-26

General and Revenue Secretariat — File No 92 of 1926

Serial No.

From Ref. Rangachari
No. B 532 dt 7-9-27.
states that no reply has been
received from E. E. Dutt
--- asked for orders.
29
9.9.27

I am afraid this is not
very much to be gained by keeping this office
open. Mr. Dutt had no practical knowledge
how his "invention" as he called it for
manufacturing cyanide of the salts at
Bhadravati and his guidance were found
to be disappointing. He has left the state
and nothing further has been heard of. The
file may be closed -

Dy
10/9/27

Dy Secy)
Ge
10.9.27

Dy
10/9

12/9

GEN. & REV. SECRETARIAT
16 SEP. 1927
DEVELOPMENT BRANCH.

NOTE- Contn. Sheet

APP. F. VI. Gl.]

SUBJECT.....

.....

.....

Section Ecol

File No. 92-26

Serial No 2.

No. D. 6263 Dt 24/2/27

Diary No. of letter replied to 4070

General and Revenue Secretariat

( )

the *192*

To

The Secy. Board of Management Sugarcane Work Bhadravati.

Date of issue 2.2.27

Signed by W. Wharry

Sir

I am directed to request you to forward for the information of Govt. a report as regards the result of ~~experiments~~ the experiments, if any, so far made by Mr Dutt, about the manufacture of Cheramides & Cyanides, at Bhadravati. ~~made by Asst. E. E. Dutt.~~

List of enclosures.....

Copied by.....

Examined by.....

Shanker
WP 28/2
to Secy. Secy

over

(4078)

M.I.W. 36.

THE MYSORE IRON WORKS

Telegraphic Address:
"MYSIRON," BHADRAVATI
(MYSORE RYS.)
Codes:
BENTLEYS
Western Union Five Letter
A.B.C. 6th Edition

Bhadravati P.O.
(Shimoga Dist.) India

Ref. No. B.327/27-

12th March 1927.

The Secretary to Government
of His Highness the Maharaja of Mysore,
Development Departments, Bangalore.

Geol. 92-26-3

Dear Sir

With reference to your letter No.D.6243/Geol.92-26-2 dated the 2nd February 1927 in regard to the manufacture of chromates, cyanide, etc., I am directed to submit herewith for the information of Government a copy of Mr. Dutt's report on the experiments carried out by him at Bhadravati and copy of a note by Dr. S.S. Aiyar, our Chief Chemist who continued the experiments after Mr. Dutt left Bhadravati.

2. The results of the experiments made by Dr. Iyer have so far been disappointing and Mr. Dutt has been informed of this and he has been requested his advice on the further action to be taken.

3 A copy of the letter addressed to Mr. Dutt is enclosed for the information of Government.

Yours faithfully,

Secretary,
Board of

Report of experiments on the manufacture of Cyanides by Heloromer Process.

Chemistry of the re-actions.

This process differs from the previous processes in that, no external heat need be applied for the reaction to proceed Further sodium nitrate is substituted for sodium chloride or carbonate, which according to the other processes should be fused with cyanamide the temperature being taken up to 1200 - 1400°.

Now the re-action may proceed according to formula 1 or 2, necessary heat being supplied by the heat of re-action of

$4\ Na_2NO_3 + 5\ C \rightarrow 2\ Na_2CO_3 + 2\ N_2 + 3\ CO_2$

1. $CaCN_2 + NaNO_3 + C \rightarrow CaCO_3 + NaCn + N_3$
2. $2\ CaCN_2 + 4\ NaNO_3 + 7\ C \Rightarrow 2\ CaCO_3 + 2\ NaCN + 4N_2 + 3\ CO_2$

The re-action is not of course so simple and does not proceed to completion only in this way. There are so many side reactions and possibly the re action is also reversible so that a certain equilibrium is established between the reacting substances and the products. Apparently $Ca(CN)_2$ is first formed which is converted to NaCN in presence of sodium salts. If all the $Ca(CN)_2$ formed does not go into NaCN, then there will be a loss of CN by the hydrolysis of $Ca(CN)_2$ in presence of water used to extract the mass.

Thus the yield is dependant on the various physicial and chemical factors which require careful control. Though there is a possibility of obtaining two molecules of NaCN from one of cyanamide yet owing to the facts that it is hard to induce carbon to combine with the second nitrogen atom of cyanamide, the calcium cyanide itself is unstable, the reaction $CaCN_2 + C \rightleftarrows Ca(CN)_2$ is reversible

and there are a number of side reactions which interfere with the main reaction, the yield is no-where near two molecules. In fact in our experiments it was found that the use of 2 molecules of nitrate to one of cyanamide renders the reaction too violent to control with the result that some cyanogen burns off seriously affecting the yield. Thus it seems that only certain maximum yield is possible under certain optimum conditions, this maximum yield lying between one and two molecules of NaCN for one molecule of $CaCN_2$. Our efforts were directed to determine these conditions.

Experiments:-

In the earlier experiments conducted here, the yields were not anywhere near that for even formula No.1. By varying the proportions of the reacting chemicals and careful control of the process it has been possible to obtain yields very near to one molecule of sodium cyanide# from one molecule of cyanamide.

Further attempts to increase with only the three chemicals, cyanamide, chilisalpeter and charcoal did not prove successful. Hence it was thought that carbide carbon might work better and so experiments with certain proportions of calcium carbide in the mixture were tried. In a small scale experiment it was actually found that an yield higher than one molecule of NaCN for one of cyanamide was obtained. This is a very interesting development, which if worked out to a successful issue may help to increase the yield and justify a patent being taken. Owing to limited time before me, I have not been able to study this further nor the influence of the various factors on the nitrogen efficiency. It has so far been not possible to get more than 52% of the cyanamide nitrogen as cyanide by this process.

Extracting Sodium cyanide.

After the reaction is over, it is necessary to cool the mass rapidly and extract the fused mass with water. By using the weak second and third extracts for treating fresh mass, it is possible to obtain solutions containing over 10 grams in 100 cc. water or say about one pound NaCN in a gallon solution. The concentration of this solution is another problem. The cyanides have the property of hydrolysing in solution and by boiling such solutions cyanogen is lost.

A loss of about 25% of the total CN in solution was obtained in these experiments the products obtained being a solution containing over 5 lbs. NaCN per gallon of solution and a wet acid containing about 30% NaCN. In more dilute solution say of 2 to 3. lbs. NaCN in a gallon, is saleable, the loss may be less. The loss is higher the more dilute the solution evaporated.

Experimental results & cost calculations.

The proportions of the raw materials which have given best results are as follows:-

Calcium cyanamide	18 parts	Commercial 19% nitrogen.
Sodium nitrate	12 "	Commercial 96% $NaNO_3$
Charcoal	3 "	Braize 56% F.C.

Yielding 6 parts sodium cyanide.

This proportion has given yields a little over the theoretical according to the formula 1 (P.1). Allowing for the losses in extraction and concentration, which as already stated come to about 25% even for a concentration into a solution of 5 lbs. in a gallon,

we can expect 75% of the theoretical. On this basis the following quantities of materials costing Rs1304/- are required to give one ton of sodium cyanide.

Cyanamide	4 tons	Rs 202/-	Rs 808
Sod. nitrate	2 2/3 "	200	466
Charcoal	2/3 "	45	30
			Rs 1304/-

(Cyanamide costs Rs162 at Bombay per ton)
(Sod. nitrate " 190 Madras ")

It is possible that the costs of the raw materials and freights etc. thereon may be lower on large scale purchases and transport The prices taken for calculation are those paid for these materials by the Mysore Iron Works in connection with these experiments.

As sodium cyanide is sold in the market on the basis of 100% KCN, we get one ton of NaCN evaluated on this basis from three tons cyanamide so that the cost per ton will be three-fourths of that calculated above i.e., Rs978/- To this must be added the cost of manufacture etc., which I expect will not be less than Rs75 per ton. Thus the cost price of sodium cyanide will be Rs1053 per ton.

Samples of Products.

A few pounds of cyanide - both as solid and solution - of the quality above mentioned, are on hand for being sent to the users thereof. These contain as impurities, ferro-cyanide, sulphide sodium carbonate and sodium hydroxide.

The first two impurities are formed owing to the presence of sulphur and iron as impurities in the cyanamide itself. These partly account for low yield of cyanide of sodium. Sodium carbona

is formed due to the action of charcoal on sodium nitrate which is the reaction responsible for generation of heat.

These are some of the side reactions already referred to, which prejudice the yield of the required product.

Financial Aspect.

Sodium cyanide - 100% KCN basis costs according to technical Journals, 7 pence a pound in England (for export). At present rate of exchange this calculates to Rs871/- per ton in England. In America sodium cyanide, 96 - 98% pure, is quoted at $446.00 per ton or Rs1252/- at current rate of exchange.

These figures are lower than our calculated cost of manufacture and our product will be mostly as solution, if the process loss should be a minimum. So I fear we have not much chance of competing with foreign products.

But the statistics of cyanide consumed in Kolar Gold Fields (vide Mr. Dutt's note) show that Rs1050 per ton have been paid in 1925. If this value could be obtained for our product on the basis of 100% KCN, the industry may be a profitable one.

On the whole, the results are not very encouraging.

Bhadravati)
1st February 1927)

Sd/- S. S. Aiyar.

Copy

THE MYSORE IRON WORKS
BHADRAVATI

12th March 1927.

My dear Mr. Dutt,

Further experiments have been carried out by our Chief Chemist Dr. Aiyar on the manufacture of chromates and sodium cyanide according to the instructions left by you but the results have so far been very disappointing.

The chromite ore does not give any appreciable yield due to the following circumstances:-

a) The reaction mass melts permitting the ore to settle down and thus to get out of the sphere of action.

b) Even with an insulation of two $1\frac{1}{2}$" thick asbestos lagging the heat does not sustain and the reaction stops in about 15 minutes.

The modification of the process i.e., fusion of the ore with caustic soda and nitre was also tried but the settlement of the ore again prejudices the efficiency of the process and necessitates heating for long time with constant stirring. Even under these conditions the yield has not been high enough to offset the high cost of the chemicals used. Further, this is hard on the crucibles which corrode very fast under the influence of heat and alkali and fuel is an additional item of expenditure.

Regarding the cyanides, the position is no better. From one molecule of cyanide it has not been possible to get more than a

[M. I. W. 21

MYSORE IRON WORKS
BHADRAVATI

molecule or a little over of sodium cyanide and there is a heavy process loss in evaporating the aqueous extract. These inflate the cost of manufacture. We get only a solid of low Na.Cn content (say 20-30%) and a solution containing 2-31 lbs. Na-Cn per gallon. Further, there are impurities such as ferro-cyanide, sulfide, carbonate and hydroxide which may prejudice the marketability of the product.

Under these circumstances, we await your advice on the matter before we proceed further.

Yours sincerely,

[illegible]

'Copy'

[illegible]

... in connection with letter of 16th inst. am enclosing?

THE MYSORE IRON WORKS
BHADRAVATI

13

[M. I. W. 21

1527

16/4

Report on experiments made at Bhadravati.

The Managing Director,
Mysore Iron Works,
Bhadravati.

GEN. & R... BRANCH.
16 APR. 1927
DEVELOPMENT BRANCH.

Dear Sir,

In accordance with our arrangement, I arrived at Bhadravati on the 20th instant and have carried out a series of experiments on the manufacture of chromates from your chrome ores and also of cyanide of soda from cyanamide, and Chilli Saltpetre. Although these -- experiments are not yet complete and certain, data I require are not available, as requested, I am giving you the following preliminary note together with my suggestions of what should be done to complete the experiments and obtain the results you require as to cost of manufacture, yields etc., on a semi-industrial scale.

Before giving you the results of the experiments, I feel that I should make the Patent position clear. It is in the interest of both the State and myself that Patent applications should be immediately filed in British India and in Mysore to protect these processes. I have received a copy of the Mysore Patent Regulations and have drawn out a specification which I hope to submit to the Secretariat at Bangalore tomorrow. At the same

time, I intend sending an application to the Controller of Patents at Calcutta for the British Indian Patent.

Description of experimental work done:

Complete analysis of the chrome ore supplied has shown it to have the following composition:-

SiO_2	3.43%
FeO	12.2 %
Al_2O_3	6.02
Cr_2O_3	52.49
CaO	1.25
Mgo	14.52
Mn	?

On treatment by the process of Helbronner and Pipereaut, it was found that this ore did not give satisfactory results, owing probably to the large amount of manganesium present. A number of mixtures were tried, the proportions being varied, but the yields in all cases were not satisfactory. Therefore, a modification of the process has been made by which the ore is to be fused with caustic soda and nitrate. The Nitrate may be either the potassium or sodium salt depending on whether it is desired to produce potassium or sodium chromate. The best result obtained by this means has been a yield of approximately of 75% of the theoretical

amount obtained with a small quantity in the Laboratory. A larger experiment is now proceeding and a further note will be submitted on this subject before my departure on Sunday.

Owing to the high cost of sulphuric acid at Bhadravati, experiments were made with a view to utilising some cheaper material in its place, for the conversion of chromate to bi-chromate. It has been found, as a result of these experiments, that the - - chromates produced can be converted into bi-chromates by means of carbonic acid gas, which is available at your works as a bye-product from your lime kilns and the products of combustion of your furnace just after being burnt under you boilers. Therefore, it would not cost more than the amount to be spent for pumping it through the solutions etc. This process is original and should be patented. A Patent specification will be drawn out shortly and sent to you. It is my intention also to apply for a Patent in British India and other countries. As this process was worked out by me in your Laboratory, the rights for the Mysore State, I will assign to the State or to the Mysore Iron Works, as directed.

THE MYSORE IRON WORKS
BHADRAVATI

I would suggest that experiments be continued in your Laboratory and also in the plant on an increasingly large scale and that data should be collected by your Chief Chemist as to the result of such experiments. I am leaving all necessary instructions for this with the Secretary of the Works with the request that he may forward a copy to you. I would further suggest that you send about one hundredweight of your chrome ore to France for treatment in the Works that are at present using the process. I could arrange that they should treat this ore and furnish you with a report of the results.

Cyanide: The process of Helbronner and Piperaaut has been adopted to the manufacture of sodium cyanide from Cyanamide. Calcium cyanamide is readily obtainable and costs Rs.165/- per ton F.O.R.Bombay. The price delivered at Bhadravati should not exceed Rs.180/- per ton The other raw material required for this process is sodium nitrate or Chilli Salt petre. It is also available and is generally of uniform quality. This material costs Rs.175/ per ton F.O.R.Bombay and Rs.190/- per ton, delivered at Bhadravati or other places in the Mysore State.

An experimental mixture of sodium nitrate and cyanamide gave a yield of about 67% of the theoretical quantity of sodium cyanide and it is hoped that this -- yield will be increased to about 80% by improvements in

the proportions of the mixtures etc. The process consisted in igniting a mixture of sodium nitrate and calcium cyanamide together with a small quantity of charcoal. From the fused mass, sodium cyanide can be leached out by dissolving in water. A solution of sodium cyanide is obtained by this means, which can be filtered from the charcoal, calcium carbonate etc., and used directly for the extraction of gold. It is suggested that solid sodium cyanide need not be prepared but that a solution of this material might be prepared in the neighbourhood of the Kolar Gold Fields and supplied to the Gold Mining Companies. The following is an estimate of the cost of one ton of sodium cyanide based upon a yield of 80% of the theoretical amount and working on cyanamide containing 19% of nitrogen as the material bought in Bombay was shown to contain. The following is an analysis:

Cyanamide.

Nitrogen.	19%
Corresponding $CaCn_2$	44.7%
Total insoluble	15.00
Free carbon.	12.50
SiO_2 etc.	--
Residue on combustion of insolubles.	2.50

THE MYSORE IRON WORKS
BHADRAVATI

Cost of producing one ton of sodium cyanide ~~is~~ in solution based on figures obtained by small scale experiments-

2.4 tons sodium nitrate at Rs.190 per ton	Rs. 380/-
2 tons cyanamide at Rs.180/- per ton.	360/-
1/4 ton charcoal at Rs.48/- per ton.	12/-
Supervision, wear on apparatus, cost of extraction by water, filtering etc.	50/-
Total Rs.	802 or
say Rs.	800/-

The solution of sodium cyanide obtained will not contain any interfering substances and should find a ~~r~~ ready market in the Gold Companies. The follwing are statistics of the quantities and value of cyanide -- consumed in the Kolar Gold Fields for the past eight years:-

Year:	quantity in lbs.	Cost £.
1925	788,348	35,485
1924	725,980	33,884
1923	678,041	35,565
1922	677,417	43,167
1921	587,568	33,545
1920	592,276	32,114
1919	781,584	44,415
1918	807,163	48,445

It will be seen that the price paid during the year 1925 worked out at about Rs.1,350/- per ton of cyanide. This leaves amargin of more than 500 per ton

ton xx when compared with the estimated cost of production. I would strongly recommend that as soon as the patent specifications are being properly filed and a few more experiments carried out with larger quantities of material by your Chief Chemist, that the Mining Companies should be asked if they would be prepared to take a solution of sodium cyanide in place of the dry salt on a unit basis of the sodium cyanide contained therein. In the event of their being prepared to do so, the manufavture of this substance should be started immediately. No plant will be required beyond a few iron crucibles, some sheet iron vats and a small filter press. Fuel is not required either for the main re-action or for the evaporation of solutions, therefore, the cyanide may be prepared at any place convenient.

Manganates: Manganese-dioxide from the Shimoga District has been alalysed with the following result:

SiO2	0.79
Al_2O_3	0.11
MnO_2	90.77
Fc	2.50
P.	0.106
Available oxygen.	16.69

THE MYSORE IRON WORKS
BHADRAVATI

As will be seen, the ore is of very high grade and is suitable for the manufacture of manganates.

I have not had time to make experiments in this direction but I am leaving instructions which will enable your Chief Chemist to carry out the necessary experiments and to report later on the treatment of this ore for the production of potassium permanganate.

Bhadravati,
31st Decr.,1926.

Sd/- Eric E.Dutt.

"Copy"

Note containing instructions for continuation of experiments etc. at Bhadravati.

Chromates: I recommend that the Chief Chemist continues the experiments on the treatment of chromite with a mixture of caustic soda and potassium or sodium nitrate at the point of fusion in cast iron crucibles. The proportions of materials which should be taken are those used for the last experiment and mentioned in his notes under the headings of charge No.20. It is thought that if the heating is continued for sufficient length of time, a good yield of chromate will be obtained by this method.

When solutions of chromate have been obtained from the ore by leaching the melt produced as above, this should be evaporated to different concentrations with a view to ascertaining the most favourable point at which to crystalise out either sodium or potassium chromate or to pass carbonic acid gas through the solution with a view to producing dichromate directly and subsequently crystalising this out from the solution. In the case of the potassium salt, it has been observed that when carbonic acid gas is passed through a concentrated solution, potassium dichromate crystalises out, the solubility of this salt being very much less than that of the corresponding chromate. On the other hand, if it is desired to produce sodium dichromate, the reverse is true. Sodium bicarbonate will be precipitated from the solution and this will have to be filtered from the bicarbonate and further concentrated before crystalising out the dichromate.

These experiments should be carried as rapidly as possible and a number of charges should be taken, the duration of heating etc., being varied according to experience gained. As soon as the results are available, they should be communicated to me and I will study the figures submitted and write a further report on receipt of the same.

Cyanide: To arrive at a conclusion regarding the cyanide experiments, I would suggest that a number of charges be prepared based on proportions of charge No.6 and maintaining a considerable excess of charcoal. The result produced by ignition of these charges should be leached with water and solution examined, data being collected as to the yield of soluble sodium cyanide.

The sodium cyanide solutions obtained by this means should be concentrated to a point were most of the foreign substances (carbonate of soda, sodium chloride etc), separate out and then diluted until they contain approximately 25% sodium cyanide. I would suggest that a solution of this composition be then submitted to one of the Gold Mining Companies at Kolar for their opinion as to whether they can use it in their extraction plant.

All results obtained should be communicated immediately to me and I will correspond with the Works regarding improvements etc., which may occur to me.

General.

Please send me at Nagpur about 20 lbs. of chromite ore and

the same quantity of manganese dioxide ore by parcel post. I have facilities for carrying some further experiments and will report as to the results.

An English translation of the French Patent is herewith submitted and I wish to have this sent to the Secretariat at Bangalore together with the necessary forms etc. If it is required that I should sign these forms, kindly send them to me at Nagpur and I will complete them and return them directly to the Secretariat.

A patent specification will be drawn out embodying the process for the conversion of chromate into bichromate by means of carbonic acid gas which was worked out in the Laboratory during my visit. The application in Mysore should be filed either by the Mysore Iron Works or by some officer of the Government, as desired I will submit a copy of my application for Patent in British India regarding this process, in due course.

Please submit a copy of this note to the General Manager, for his information.

Bhadravati,
2-1-1927.

(Sd) E. E. Dutt.

Copy

Chromium oxide (Cr_2O_3)

Oxide of chromium is used for the preparation of a number of chromium compounds, also for the production of pure chromium metal by the Thermit process. Owing to the development of chromium plating in America and Europe, considerable demand has arisen for this substance.

Chromium oxide is easily prepared either from chromate of soda or potash by heating with charcoal or by igniting a mixture of alkali chromate and charcoal, and extracting the alkali carbonate formed by water.

It is estimated that pure chromium oxide can be manufactured at Bhadravati for about Rs.700/- per ton. The oxide is at present quoted at 1 sh 2d per lb. in London.

If desired, I can communicate with my friends in America and ascertain the quantities of this material they can dispose off and approximately at what figure

---

MYSORE IRON WORKS, BHADRAVATI

SPECIFICATION.

Improvements in processes for carrying out chemical reactions.

Up to the present, it has been necessary when carrying out dry chemical reactions where heat is required to expend considerable quantities of energy, the major portion of which is lost in heating the lining (walls) of the furnace, crucibles, muffle, etc. in which the operation is conducted.

If on the other hand, it is desired to submit the reacting mass to the action of a vapour, (as for example, to the action of oxygen in the case of an oxidisation process), it has been found difficult to keep the gas in intimate contact with the aggregate of the materials undergoing treatment.

The present invention obviates these difficulties by means of an internal heating "in situ" of the materials to be treated.

The process consists in employing an explosive reaction, the rapidity of which is greatly retarded; by this means, it is possible to obtain a sort of combustion of the explosive and the heat of decomposition is no longer converted into a balistique effect but is utilised for producing a heating of the reacting materials upto fusion, if necessary. In order to diminish the velocity or rapidity of the explosive reaction, it is only necessary to mix the explosive substance with the other components entering into reaction; this inert mass acts as a diluent from the point of view of the propogation of the explosive wave.

Several examples may be given:-

1. The explosive reaction may be obtained as an integral part of the operation, it is desired to carry out.

2. Together with the explosive reaction, it may be advantageous or necessary to employ complementary exothermic reaction, the initial high temperature being produced by the explosive reaction and afterwards being maintained by the complementary reaction. For example, air may be blown into a mass to which carbon has been added.

3. The operation may require the employment of a gas in which case this is blown through the reacting mass; in the case of an oxidisation, for example, air, preferably preheated, in order to reduce to a minimum the lowering temperature of the mass undergoing reaction.

4. The operation, it is desired to carry out, may require a combination of 1 and 2 or 1, 2 and 3 together. In order to further illustrate the application of the invention, the following examples have been given:-

Examples:- 1. Black powder as an explosive and carbonate of soda as the diluent. As in the case of gun powder, which is a granular and homegenous mixutre, a mixture of potassium or sodium nitrate, carbon and sodium or potassium carbonate is prepared. The following proportions may be mentioned. Sodium nitrate, 75 parts or equivalent quantity of potassium nitrate. Carbon 18 parts, sodium carbonate 40 parts. Ignition is effected by means of a mixture of sodium chlorate or barium peroxide, or the like, with magnesium powder. This is lighted with magnesium ribbon. The

following reaction occurs:-

$$4NaNo_3 + 5C = 2NaC_2O_3 + 3Co^2 + 2N^2$$

By this means large quantities of sodium carbonate may be obtained in a fused state in an iron receptacle, an operation hitherto impracticable on an industrial scale. The iron is protected externally by a refractory coating and resists perfectl the action of the alkali.

It is possible to carry out all alkaline attacks on minerals in this manner. For instance, an alkaline fusion may be made to obtain uranates, vanadates, chromates, manganates, zincates and the like. The elevated temperature necessary for the reaction is obtained by the explosive reaction in the presence of sodium carbonate and the mineral. To the fused mass of carbonate of soda and mineral carbon may be added and preheated air blown in to maintain the temperature at the point of fusion by the oxidisation of the carbon and also at the same time to oxidise the mass in reaction if necessary as in the case in the treatment of sesquioxides.

It is possible to give a number of examples, among which the caustic ising of sodium carbonate may be mentioned. To carry this out, a mixture of sodium nitrate, c rbon and carbonate of soda is placed in the centre of an iron vessel containing carbonate of soda and caustic lime (CaO). A cartridge is placed in the mixture of sodium nitrate, carbon, etc., composed of potassium chlorate and magnesium powder and this is ignited by means of magnesium ribbon. When the whole mass quietly fuses, the mixture is stirred, a certain quantity of carbon being added and sulfi

sufficient air blown in to burn the carbon and maintain the whole in a state of fusion. The calcium carbonate formed will be dissassociated to a certain extent, owing to the elevated temperature but the carbon dioxide liberated is without action on the soda as it is in the form of Na_2O. In an analagous manner, the following reaction may also be carried out.

$$Ca_3 (Po_4)^2 + 3\ Na_2CO_3 = 2Na_3\ Po_4 + 3CaCo_3$$

By this means, sodium phosphate is produced and this can then be readily converted into phosphoric acid.

This process may equally be adopted to the treatment of complex ores, in particular, to the treatment of sulphide ores such as, zinc blend galena and the like. Taking for example the case of zinc blend, this may be treated with sodium nitrate. It is not necessary in this case to add carbon and sulphur as these are replaced by the reducing action of the sulphur and zinc in the ore, which are both oxidised and yield a stable zincate, which is soluble in water and can be separated from the other constituents of the ord by this means.

Any other explosive mixture may be employed in place of the black powder described above and the carbon may be in any form free or combined, as for example, carbon in the form of carbides.

Claims.

1. A process for carrying out chemical reactions by means of an internal combustion, which consists in employing an

an explosive reaction, whose velocity is greatly retarded by mixing the explosive substance with the whole mass of the material entering into reaction.

(a) The explosive reaction may itself effect the operation desired.

(b) A complementary exothermic reaction may be employed concurrently with the explosive reaction.

(c) A gas or air may be blown into the reacting mass.

The different processes indicated in a, b and c may be employed in combination.

2. Application of the process to alkaline fusion and attack of minerals with oxidisation, as for example, the oxidisation of Sesquioxides, or without oxidisation as in the case of the dry caustification of sodium carbonate for the production of sodium phosphate and treatment of complex ores.

3. A process by which chemical reactions may be effected by an internal heating of the reacting masses brought about by an explosive or highly exothermic reaction substantially as herein described.

Copy

MYSORE IRON WORKS, BHADRAVATI

Experiments on the manufacture of chromates by the process of Helbronner & Pipereaut.

Process.

According to this process the ore is to be intimately mixed with an explosive mixture and fixed, the latter giving the heat necessary for the reaction as also the oxygen for the oxidation of chromium sesquioxide and the potassium for its conversion to potassium chromate. The materials used in the experiments were:-

Chromite ore from Thiptur area;
Potassium nitrate (Bengal nitre) from Best & Co.
Charcoal braize of the Plant.

These were crushed, sieved and graded as per instructions of Mr. Dutt.

Experiments.

Several charges were taken the proportion of the materials being based on the molecular ratio and the purity of the substances, giving a little excess over the theoretical amounts. The best result obtained was only about 10% of the theoretical yield, and this in an experiment where sulphur was also used in addition to charcoal, to increase the intensity of the reaction. In almost all the cases the reaction was at an end in

about 10 to 15 minutes and the mass left behind contained much of unacted ore, potassium carbonate, nitrate , nitrite etc. In order to conserve the heat of reaction, the crucible containing the mixture was insulated with a double wall of asbestos. Yet the yield in no way improved. The unacted ore was invariably found to have settled to the bottom of the crucible.

Reasons for this behaviour.

Chromite is a refractory mineral and requires even under best conditions of oxidation treatment with sodium per-oxide in the laboratory, longer period heating than that obtained in the above experiments. In these experiments, the oxidising chemical used was crude potassium nitrate (73.6% pure) which melts at 337°C and decomposes at 400o Owing to the explosive reaction between nitrate and charcoal the mass melts and the chromite being heavy, settles to the bottom, thus actually getting out of the sphere of action. The nitrate itself gets partly converted nitrite, carbonate, oxide, etc. which are less active.

Process modification suggested by Mr. Dutt.

Mr. Dutt suggested fusing the ore with potassium nitrate and sodium hydroxide. Five charges were taken with proportion of materials required for complete conversion of

Cr_2O_3 to chromate, based on molecular ratios and the purity of the materials. The results are tabulated below:-

Charge No.	Yield percent of K_2CrO_4 over the theoretical	No. of hours heating at low red heat.
42	About 17%	1
43	38.4%	2
44	Crucible sprang a hole at the bottom in 6 fusion hrs.	
45	36.8	2-45 minutes.
46	59%	4

In the actual recovery of the chromate there is a process loss of about 5% in handling and in what the ~~mixxxxxx~~ mother liquor retains. The latter however, is not entirely lost as it can be recovered by drying up the liquor and using the alkali cake containing nitrite, carbonate hyxroxide with a little chromate, for the following fusions provided the oxygen deficiency can be made up by atmospheric oxygen Air will thus have to be blown through the mass. This can best be done if the mass can be kept porous and nonfusible by a little modification of the process to be discussed later.

Interpretation of the results.

Even in the fusion method it is, I think, not possible to get satisfactory results. For, on the one hand,

the ore settles to the bottom thus escaping the rapid action of the chemicals and on the other the chemicals themselves volatilize, the more so the longer we heat the mass. These factors operate against the reaction going satisfactorily, with the results that the yields cannot be satisfactory.

Furthermore, this process seems to have a destructive action on the iron crucibles used. One 12" foundry ladle used for this purpose, made of ¼" thick sheet, sprang a hole at the bottom in four fusions aggregating to 6 heating hours. This is another factor against this process, as it means a heavy depreciation on the crucibles used for fusion.

In this process part of chromate is in the form of potassium salt and part as sodium salt. On concentrating the extract, most of chromate crystallises out, the sodium salt tending more to remain in solution. On further concentration the chromate comes with much of the impurities - e.g. nitrate, carbonate, etc - in solution, so that the purification process will be tedious. It is best to use only potassium or sodium nitrate and hydroxide to render separation and crystallisation easier.

Unless the reaction goes to completion it will be advisible to separate the chromate first and convert it to dichromate istead of going direct to dichromate to avoid the interference of the impurities and reduce the quantity

of sulphuric acid to be used.

Financial.

The best results so far obtained has been only 59% of the theoretical yield. Hence for one ton potassium chromate the following materials are required:-

1. Chromite 1.33 tons @ Rs25 per ton	Rs 42/-	Mining Superintendent's estimate. I think this is rather low.
2. Nitre 1.33 tons @ Rs300 per ton.	400	Stores value Prices paid by Iron Works for the materials actually purchased.
3. Caustic soda 8 cwts. @Rs400 "	160	
4. Potassium sulphate 8 cwts @Rs200	80	Based on market quotation in English Journals.
5. Or in place of 3 & 4 we may use 8 cwts potassium hydroxide @ Rs560 per ton	224	Do. allowing for freight, etc.

It is cheaper and more efficient to use potassium hydroxide instead of items 3 and 4 and hence I take the valuation using items 1, 2 and 5. This brings the cost of one ton chromate to Rs666/-. To this must be added the charges for supervision, labour, coke (for fusion), steam (for concentrating the extracts) depreciation on plant especially crucibles which I think will not less than Rs84/- This brings up the manufacturing price of potassium chromate to Rs750.

If this is to be converted to dichromate the cost of dichromate works out thus:

4 tons chromate gives about 3 tons dichromate taking about 1 ton sulphuric acid. There is a further process cost on labour, steam, etc. Thus 1 ton potassium dichromate will cost not less than ₨1200 a ton. But according to the import statistics the cost per ton of dichromate in 1924-25 was ₨662/- only By heating for a longer time say 5 - 6 hours, I think it may be possible to get higher yields. Even assuming we get up to 80% (above which it is not possible to go for reasons already mentioned), the cost per ton will be ₨900/- which is yet too high to permit of competition against imported materials.

Calculating on a similar basis sodium chromate cost ₨500 a ton and sodium dichromate about ₨680 allowing for process loss, etc. Based on 80% yield sodium chromate cost ₨375 and dichromate ₨660. These prices are again higher than the import prices, sodium dichromate having been imported at ₨484 in 1924-25. It is possible to get the chemicals especially sulphuric acid, cheaper than the values here assumed, on wholesale purchase and freight basis Yet I fear we may not reach down to the present market rates.

Suggestions.

In view of the fact that according to neither of the

two processes above referred to, the chromite ore is efficiently and profitably converted to chromate, I believe, it would be advisable to try the existing process of heating the ore under non-fusible condition exposed to atmospheric oxygen (i.e. a strong current of air being passed over the reaction mass). It is said that this process gives very satisfactory yields and requires only cheap chemicals like lime, ~~potash~~ sodaash and potassium chloride or sulphate the last only if the sodium dichromate obtained is to be converted to potassium dichromate.

Conclusions

(1) The process of Helbronner and Pipereaut does not work satisfactorily for the conversion of chromite ore into alkali chromates. It would be advisible to get all the details available from the authors regarding the working of the process and the plant to be used therefor before further experiments are undertaken.

(2) The fusion method suggested by Mr. Dutt is also not very satisfactory. With the yield results so far obtained the costs of potassium chromate and dichromate are much higher than import rates.

(3) It is suggested that experiments according to the existing processes may be tried. According to literature yields are higher by these processes and chemicals used less costly.

Bhadravati.)
15th February 1927)

S. S. Ayar

APP. F. VI. Gl.]

SUBJECT.....

.....

.....

Section Ecdr

File No. 72 - 26

Seria No. 4

No. P-8230 dt 26/3/27

Ecdr

Diary No. of letter replied to.....

To The Dir. of Ecdr

.....

General and Revenue Secretariat

( )

the *192*

Sir

Date of issue 28.3.27

I am directed to forward for perusal and return reports received from the Assessors: E.E. Dutt & S. S. Iyer regarding the manufacture of Cheroots, Cigarettes &c

Signed by [illegible]

I have &c

WD

23/3

for Dep. Secy

List of enclosures.....

Co by.....

Examined by.....

over

M.G.D. 12.] *Dated, Bangalore,* 12-6-1927

No. D2 Dis 12/26-27 Encl

GEN. & REV. SECRETARIAT 6768 14 APR 1927

FROM

P. SAMPAT IYENGAR, ESQ., B.A., B.L.,
Director, Mysore Geological Department.

Ref 92-26-5

N. Rama Rao, Esq., B.A., B.L.,
Secretary to the Government of
His Highness the Maharaja of Mysore,
Development Departments.

SIR,

I have the honour to ~~forward~~ return herewith the reports of Messrs. E.E. Dutt, and S.S. Aiyar, regarding the manufacture of chromates, cyanides, etc. received with your letter No. D.8230/Geol.92-26-4, dated the 26th ultimo,

I have the honur to be,
Sir,
Your most obedient servant,

P. Sampat Iyengar

Director,
Mysore Geological Department.

M.R/12.

62

APP. F. VI. Gl.]

SUBJECT.....

.....

.....

Section Ecl

File No. 92-26

Serial No. 1

D No. D-2615 of 1927/27

Diary No. of letter replied to.....

General and Revenue Secretariat

( )

the *192*

Date of issue 15-9-27

Signed by .....

List of enclosures.....

Copied by.....

Examined by .....

To

The Secy. Board of Management, Improvement Works

Dear Sir

In inviting ~~your~~ attention to your letter no. B 327/27 of 12-3-27 in the matter of experiments made ~~about the~~ by me. Both about the manufacture of Chromates & Cyanides. I am desired to request you to forward for the information of Govt. a final report about ~~the~~ ~~and Both as to~~ the action taken by you in the ~~matter examined~~

over

THE MYSORE IRON WORKS
BHADRAVATI

44

No. B.532/27

7th September 1927.

DEVELOPMENT BRANCH.

Dear Sir,

With reference to your D.O. letter No. D.2419/Geol.92-26 dated 5th September 1927, I am to state that no reply has been received to the letter of 12 March 1927 (copy sent with this office letter to you of 12.3.1927) addressed to Mr. Eric E. Dutt requesting his advice in regard to the disappointing results of experiments conducted by the Chief Chemist, Dr. S.S. Aiyar, according to the instructions left by Mr. Dutt.

Yours sincerely,

M. Rangaswamy

P.H. Krishna Rao, Esq., M.A.,
Assistant Secretary to Government,
Bangalore.